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FACILITY FORM 602

N65-24846 (ACCESSION NUMBER)	(THRU) 1
25 (PAGES)	(CODE) 33
CR 65015 (NASA CR OR TMX OR AD NUMBER)	(CATEGORY)

GPO PRICE $ ____________

OTS PRICE(S) $ ____________

Hard copy (HC) 1.00

Microfiche (MF) .50

NASA-CR-65015

Report Number 1675

PERFORMANCE EVALUATION OF "THERMO-LAG" MATERIAL FOR ENTRY HEAT PROTECTION OF ADVANCED MANNED SPACECRAFT

FINAL REPORT

October 1, 1962 - January 31, 1964

Volume One - Summary of Investigations

Prepared For

National Aeronautics and Space Administration

Manned Spacecraft Center

Houston 1, Texas

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INTRODUCTION

In accordance with the requirements of Contract No. NAS 9-877 negotiated by the National Aeronautics and Space Administration, Manned Spacecraft Center, General Research Procurement Office, Houston, Texas with The Emerson Electric Manufacturing Company of St. Louis, Missouri, for Performance Evaluation of "THERMO-LAG" Material for Entry Heat Protection of Advanced Manned Spacecraft, this final report is submitted to document and summarize the results of the entire contract work.

The report is submitted in five (5) volumes: Volume I, Summary of Investigations; Volume II, Thermal Investigations, Volume III, Structural Investigations; Volume IV, Blister Investigations; and Volume V, IBM Manual.

Two forms of "THERMO-LAG" T-500 were evaluated. The T-500-4 designation refers to spray-deposited material cured according to prescribed procedures. The designation T-500-6 is applied to spray deposited material, reinforced with loose weave glass cloth weighing 1.94 ounces per square yard interlaminated during the spray operations at intervals of 0.050 inches, which has been vacuum bag molded and cured according to prescribed procedures.

The report includes detailed descriptions of the investigations performed and the results obtained pertinent to the use of "THERMO-LAG" T-500 materials for the fabrication of manned entry vehicle heat shield systems. Investigations significant from the standpoint of thermal performance include the determination of thermal properties, thermal-chemical characteristics, environmental characteristics, and the ablation characteristics of T-500 materials as evaluated from the results of an extensive program of air arc plasma flight simulation tests. Basic information was derived concerning the ablation mechanism which was used for the development of a numerical computer program to predict thermal barrier requirements.

Structural investigations originally contracted include the determination of material structural properties over a range of temperatures and the performance of a simplified stress analysis of T-500 heat shield systems applied over a honeycomb structure. During the course of this work, a unique method of determining the thermal-structural behavior of a heat shield was developed involving the simulation of the heat shield continuum with a truss and shear web network. This report includes a description of the work performed under contract extension to refine and expand the truss analogy studies and to develop an IBM program for rapid problem solution.

Investigations are reported concerning "THERMO-LAG" application methods, mechanical fastener development, and the use of the material as a filler for joints and gaps. Cylindrical PH 15-7 steel - heat shield assemblies were exposed to severe thermal cycling to assess the adequacy of various attachment methods.

The report includes recommendations for the performance enhancement, and further development and testing of "THERMO-LAG" material.

PHASE I

THERMAL PROPERTIES AND PERFORMANCE OF "THERMO-LAG" T-500 MATERIAL

CONTRACT REQUIREMENTS

The Contractor will conduct a test program upon "THERMO-LAG" T-500 EX167 thermostatic subliming material to determine its thermal properties and performance characteristics and its behavior in the space environment.

Conductivity, specific heat, emissivity, coefficient of expansion, of "THERMO-LAG" T-500 shall be determined as a function of temperature both when the material is in the virgin state and in the charred state. If suitable methods can be developed, properties shall also be determined in one or two intermediate states. Rate and temperatures of decomposition shall be determined by Thermogravimetric Analysis. Overall heats of decomposition shall be measured, and qualitative studies of decomposition shall be determined by Differential Thermal Analysis. Gases produced shall be analyzed. The effect of rate of char formation, char density, etc., on these properties shall be explored.

Thermal performance shall be studied for environments typical of Apollo class vehicles. This performance shall be assessed by simulation of entry heating in an uncontaminated plasma facility. A range of enthalpy, heating rate and oxygen content shall be covered for both steady state and transient simulations. Comparative radiant lamp simulations shall be conducted.

Consideration shall be given to the effects of the space environment, and vacuum exposure upon the subsequent performance of the ablation materials.

CONTRACT INVESTIGATIONS AND RESULTS

Thermal Conductivity values were determined for "THERMO-LAG" T-500-4 and T-500-6 in both the virgin and charred states. Determinations were made over a temperature range of -150° to 400° F. in accordance with the ASTM C177-45 guarded hotplate procedure. Abridged results are as follows:

Thermal Conductivity - Btu/Hr-Ft°F.

Test Temperature, °F.	-150	0	77	250	400
T-500-4, Virgin	0.052	0.069	0.080	0.064	0.102
T-500-4, Char	0.038	0.038	0.038	0.037	0.036
T-500-6, Virgin	0.074	0.055	0.060	0.070	0.064
T-500-6, Char	0.043	0.042	0.041	0.039	0.038

Specific heats of virgin and charred "THERMO-LAG" T-500 materials were determined over a range of temperatures by a method of mixtures employing a Parr Adiabatic Calorimeter and using toluene as the immersion fluid. The interpolated results were:

Mean Specific Heat, Btu/Lb °F.

Test Temperature, °F.	-116	0	100	240	400
T-500-4, Virgin	0.23	0.26	0.36	0.37	0.60
T-500-4, Char	0.32	0.32	0.32	0.40	0.65
T-500-6, Virgin	0.20	0.25	0.31	0.40	0.56
T-500-6, Char	0.27	0.26	0.34	0.37	0.41

DENSITY. Using Method A of ASTM D792-50 and a Fisher-Young Gravitometer, the densities of virgin "THERMO-LAG" T-500 materials were determined over a range of temperatures with the following results:

Density, Lb/Ft^3

Test Temperature, °F	-100	0	77	250	400
T-500-4	67.0	52.5	62.0	59.5	58.0
T-500-6	68.5	62.5	62.0	60.0	59.5

EMISSIVITY. Total normal emissivity values for "THERMO-LAG" T-500 materials were determined over a range of temperatures above, below, and at the sublimation temperature. The determinations were made, under sub-contract, by Washington University using an apparatus consisting of an environmental guard maintained at test temperature, a reference black body heated to the appropriate temperature, and a thermopile radiometer.

Test data indicate the occurrence of marked emissivity changes during sublimation of "THERMO-LAG" T-500 materials. At temperatures ranging from circa 100 to 400° F. the emissivity of precharred "THERMO-LAG" T-500 material is approximately 0.93. Virgin materials prior to sublimation range in emissivity from 0.9 to 0.98. During sublimation, and at temperatures above the sublimation temperature emissivity values decrease with "THERMO-LAG" T-500-4 approaching 0.8 at 700°F. and "THERMO-LAG" T-500-6 approaching 0.6 at 700°F. With further temperature increase where oxidation effects are pronounced, emissivity values would be expected to increase.

LINEAR COEFFICIENT OF THERMAL EXPANSION. Determinations of thermal expansion coefficients were made in accordance with ASTM D696-44 for both virgin and charred "THERMO-LAG" T-500 materials at nominal test temperatures ranging from -140° to 315°F. These values are:

Linear Coefficient of Thermal Expansion, In/In-°F

Nominal Test Temperature, °F	-140	0	77	250	315
T-500-4, Virgin	1.7×10^{-5}	3.5×10^{-5}	2.6×10^{-5}	2.7×10^{-5}	1.8×10^{-5}
T-500-4, Char	0.9×10^{-5}	1.4×10^{-5}	1.8×10^{-5}	1.4×10^{-5}	1.4×10^{-5}
T-500-6, Virgin	1.8×10^{-5}	3.1×10^{-5}	2.0×10^{-5}	0.7×10^{-5}	0.6×10^{-5}
T-500-6, Char	0.7×10^{-5}	1.2×10^{-5}	1.3×10^{-5}	1.7×10^{-5}	0.9×10^{-5}

THERMOGRAVIMETRIC ANALYSIS. Rates and temperatures of decomposition and changes of state of the "THERMO-LAG" T-500 materials were determined by recording sample weights during exposure to controlled temperatures increasing at a constant rate. An apparatus consisting essentially of a rate controlled oven and a Cahn electrobalance with associated programmers and recorders was used. Weight versus temperature histories at three rates of oven temperature increase show the highest rate of weight loss to occur between oven temperatures of 450° and 550°F. No significant differences were observed between "THERMO-LAG" T-500-4 and T-500-6.

DIFFERENTIAL THERMAL ANALYSIS. The thermal behavior of "THERMO-LAG" T-500 materials under controlled temperature rise was studied by continuously recording the difference in temperature between a "THERMO-LAG" T-500 specimen and a thermally inert silica material during simultaneous heating. A Deltatherm DTA apparatus was used. Tests were conducted at three rates of temperature increase. For both "THERMO-LAG" T-500-6 and T-500-4, differential temperature deflections indicated two definite endothermic phenomena to occur within the temperature range 200° to 1000°F. A minor endotherm occurring at approximately 420°F. is attributed to a crystalline rearrangement of the subliming inorganic salt. A second and major endotherm indicative of the sublimation temperature of the "THERMO-LAG" T-500 materials begins at 545° to 560°F. The DTA apparatus was calibrated using $AgNO_3$, having a known heat of fusion of 16.2 cal/gm. Based on this calibration, the overall heat of decomposition of "THERMO-LAG" T-500 material was calculated at 1550 Btu/Lb.

GAS ANALYSIS. Gases produced from "THERMO-LAG" T-500 materials during ablation were subsequently analyzed using a Beckman GC-2A chromatograph equipped with silicone and molecular sieve columns. Although calibration of the silicone column was not attained for BF_3 and HF, the presence of which is predictable, approximately 87 percent by volume of the evolved gas was identified as follows:

H_2 - 45 percent	N_2 - 0.8 percent	CO - 6.6 percent	C_2H_6 - 1.4 percent
O_2 - 0.1 percent	CH_4 - 17.6 percent	CO_2, C_2H_4 - 8.2	H_2S - 5.8 percent

THERMAL PERFORMANCE OF "THERMO-LAG" T-500 FOR ENVIRONMENTS TYPICAL OF APOLLO CLASS VEHICLES

AIR-ARC FLIGHT SIMULATION TESTS. Plasma jet tests of "THERMO-LAG" T-500 materials were conducted at stagnation point heat fluxes ranging from 30 to 456 Btu/Ft^2-Sec. and at enthalpies ranging from 2500 to 23,000 Btu/Lb. Test condition matrices and model configurations were designed to obtain, from each model, material performance information relative to thick, stagnation region heat shields and thinner (0.1 inch maximum) aft body coatings. Data obtained from this extensive test effort were analyzed to provide performance information directly applicable to the design of an Apollo type vehicle heat shield and to provide fundamental definition of the "THERMO-LAG" T-500 ablation mechanism suitable for the establishment of a numerical ablation program for computer solution.

1. A transient ablation test series consisting of 45 plasma jet runs was conducted at plasma stream conditions ranging from 3500 to 23,000 Btu/Lb total enthalpy and corresponding cold wall stagnation point heat fluxes ranging from 35 to 456 Btu/Ft^2-Sec. Six conditions were selected for the series. Multiple runs were made at each condition with varying exposure time. Following exposure, test models were sectioned to permit measurement of virgin and char material recession at the stagnation point and at sidewall locations. Test measurements were used to construct, for each stream condition, plots showing recession histories, mass loss rates, and surface temperatures as functions of exposure time.

   The test results and observed "THERMO-LAG" T-500 performance characteristics fell into two general categories: The first, typified by the transient history shown in Figure 1, includes test conditions in the lower heat flux-enthalpy range where surface temperatures did not

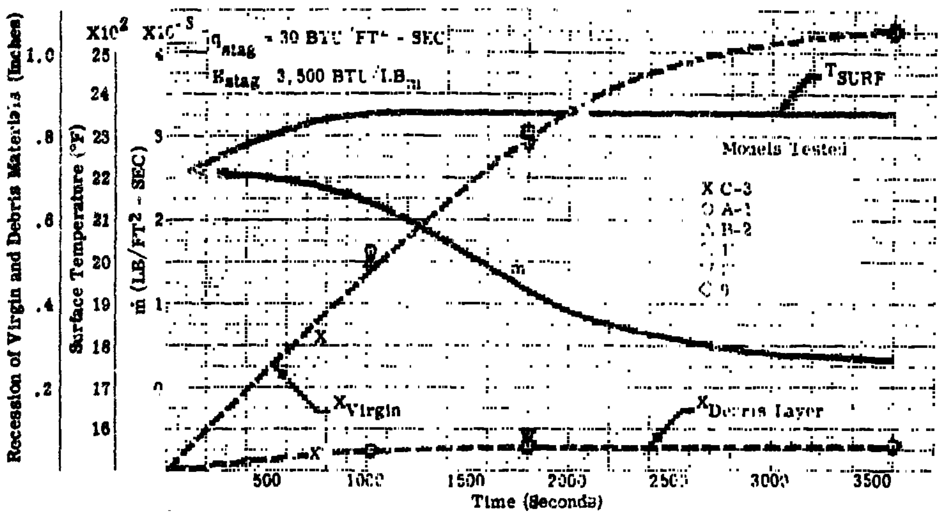

Figure 1. "THERMO-LAG" T-500 Transient History (Low Range)

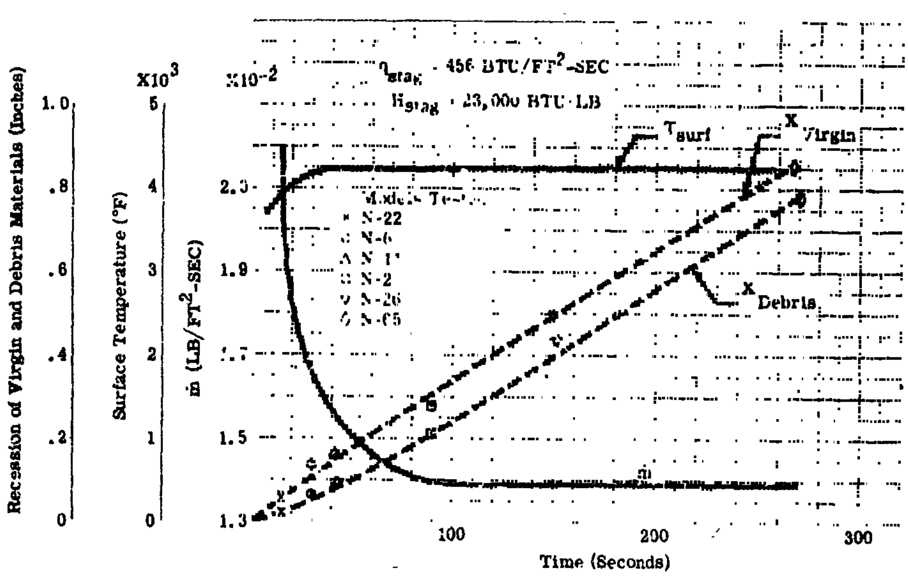

Figure 2. "THERMO-LAG" T-500 Transient History (High Range)

exceed 3100°R. Within this range of conditions, no change in model configuration was observed, recession histories were nonlinear, and the thickness of the debris layer increased throughout the test duration. At higher heat-flux-enthalpy conditions, (110 Btu/Ft^2-Sec., 10,000 Btu/Lb and above), the ablation behavior showed the effects of debris loss through oxidation. Performance was characterized by a relatively short transient phase of rapid debris layer growth to a maximum thickness followed by ablation during which a nearly constant debris layer thickness was maintained. A transient history typical of the second category is shown in Figure 2.

2. Air arc tests of "THERMO-LAG" T-500 were performed to investigate the effects on material performance of variations in the oxygen content of the high energy plasma stream. Sixteen tests were performed, at four stream conditions within the enthalpy range of 2500 to 15,000 Btu/Lb and the corresponding stagnation heat flux range of 48 to 300 Btu/Ft^2-Sec. Four tests were conducted at each stream condition at oxygen concentrations of 0, 7, 15, and 21 percent by volume. At all conditions, model surface temperatures increased with increased stream oxygen as did total material recession. A correlation of debris recession rates is shown in Figure 3. The data indicate that the surface recession rate increases exponentially to a surface temperature of about 4500°R at which point a diffusion process becomes controlling.

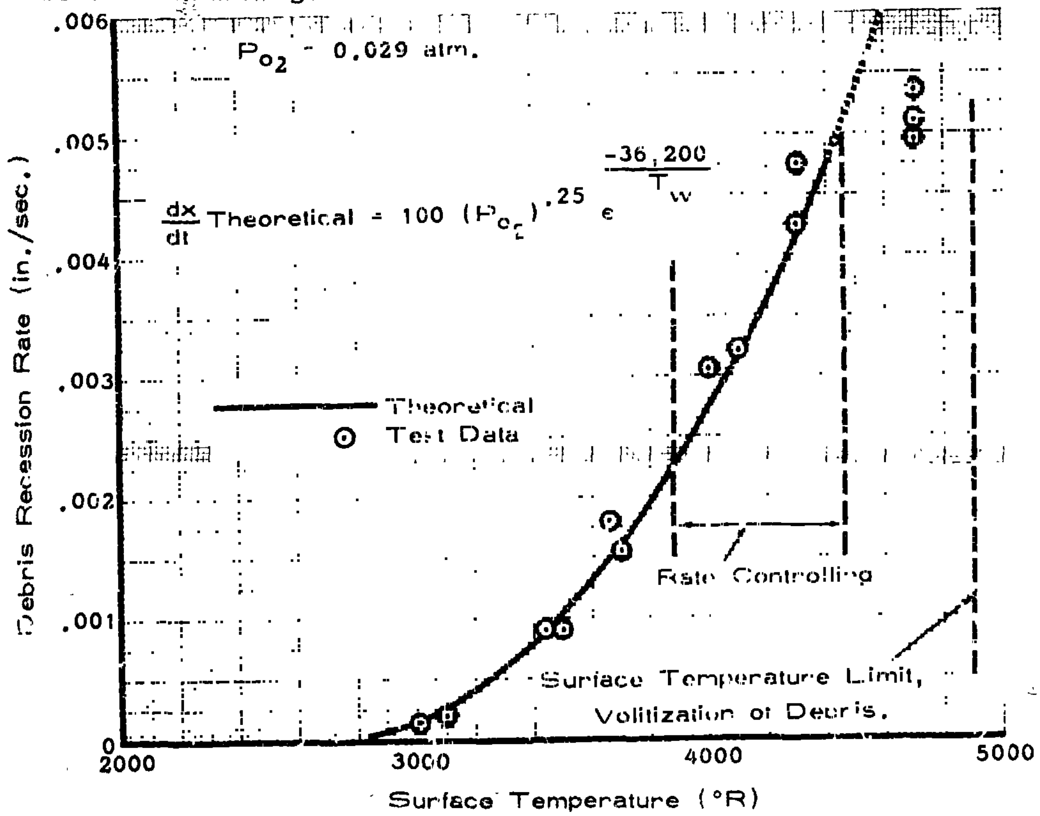

Figure 3. Correlation of Debris Recession Data

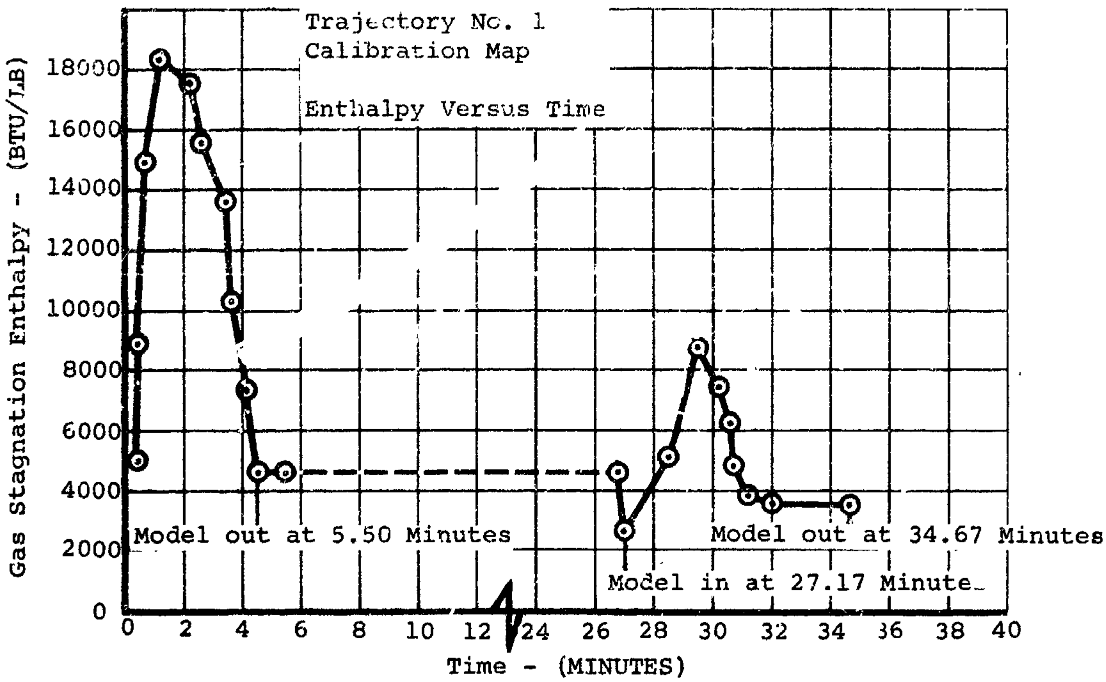

Figure 4. Variation of Enthalpy With Time for Overshoot Trajectory Simulation Test

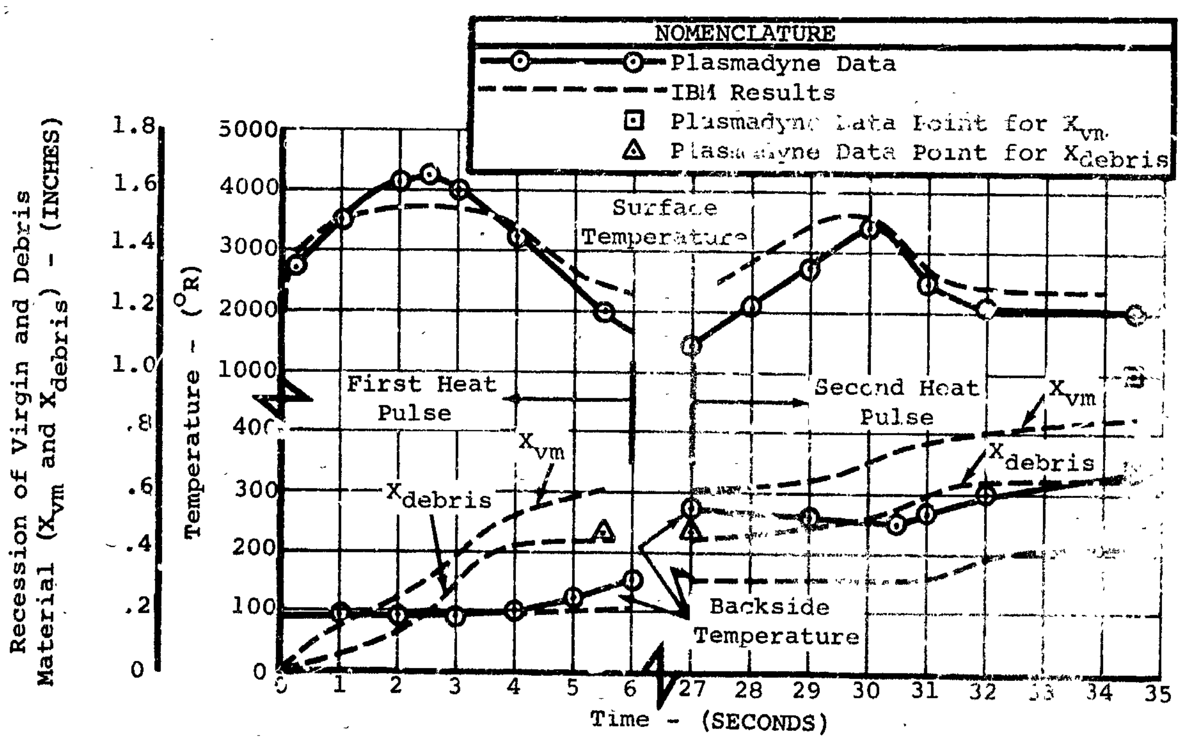

Figure 5. Comparison of Ablation Results - Trajectory Simulation.

3. Air arc tests were performed to establish the relationship between stagnation pressures and effective gross heat of ablation of the "THERMO-LAG" T-500 material. Tests were run at constant total enthalpies of 12,000, 17,500, and 25,000 Btu/Lb. Pressures were varied by a factor of approximately four at each enthalpy. It was expected that the pressure at the surface of the ablator would influence the heat transfer rate, chemical reactions at the ablator surface, oxygen diffusion to the surface, aerodynamic shear, and chemical reactions within the debris layer, all of which would affect the average cold wall ablator efficiency. Test results indicated a general increase in average gross heat of ablation with increased stagnation pressure, for example; at a constant boundary layer enthalpy difference of 10,500 Btu/Lb., a 13 percent increase in gross effectiveness resulted from an increase in stagnation pressure from 0.005 to 0.020 atmospheres.

4. An evaluation of material performance response to varying total enthalpy at constant heat flux was conducted to obtain a measure of the energy transferred within the boundary layer and to determine effects on the debris layer. The test series was designed to cover a total enthalpy range of 2500 to 17,500 Btu/Lb and heating rates of 50 to 300 Btu/Ft^2-Sec. Test results showed material performance at total enthalpies of 5000 Btu/Lb and less to be essentially independent of boundary layer enthalpy difference. Also, the absence of apparent significant oxidation reactions permitted re-radiation of most of the incident heat flux. Above 5000 Btu/Lb, the effective heat of ablation was found to increase with increased total enthalpy.

5. In the course of the air arc test program, several different model geometries were utilized to achieve a range of heat flux while maintaining constant stagnation pressure and total enthalpy. Model configurations tested included 5/8 and 1 inch radius hemisphere-cylinders, hemisphere-cones, and flat-faced cylinders. While evaluation of the test results indicated the initial model shape to have an effect on ablation performance, a complicating factor was observed in many cases where model shape varied during exposure. It can be generally concluded from the results that, for total enthalpies of 10,000 Btu/Lb and above, and for comparable surface temperatures, debris thicknesses were greater for models of larger diameter with associated greater apparent efficiency.

6. Tests were performed with the environmental heat flux, enthalpy, and low stream established as functions of time to simulate, insofar as permitted by the limitations of the test facility, the performance range required of a heat shield material for protection of a superorbital manned spacecraft during the entry maneuver. The test was designed to simulate the Apollo overshoot trajectory. Figure 4 shows the trajectory calibration map in terms of stagnation enthalpy and time. Data from the trajectory simulation test were compared with results of IBM computer prediction runs as shown in Figure 5. Trajectory simulation test results agreed with computer predictions of debris layer surface temperatures and total material recession within 10 percent.

7. An air-arc test series was performed to determine the extent to which a T-500 heat shield, interrupted by gaps of various dimensions can provide thermal protection to a substrate material. A water-cooled specimen holder was designed and fabricated to remove about 200 Btu/Ft^2-Sec. at the stagnation line. The holder accommodated two gap test specimens, one with the gap parallel to stream flow and the other

normal to stream flow. Gap dimensions were selected to be proportionately representative of heat shield discontinuities that might be expected at joints, windows, and hatches. Substrate temperatures beneath the heat shield gap and beneath the heat shield material 0.125 inch from the gap center were measured during exposure to stream conditions having total enthalpies of 10,000 and 17,500 Btu/Lb. Under the conditions investigated gap temperatures did not exceed 110 percent of protected substrate temperature, with significant temperature differences occurring only in the specimens having gaps normal to stream flow.

8. Air arc tests were conducted to obtain experimental data bearing on the effect of stagnation region ablation upon afterbody heat transfer and ablation of "THERMO-LAG" T-500 material. Two dimensional test models of 3 types were designed having cylindrical leading edges of one inch radius constructed of copper, graphite, and "THERMO-LAG" T-500. Provision was made to permit insertion of instrumented test coupons aft of the leading edge. Test models were exposed to stagnation heating rates of 36, 79.5, and 121.3 Btu/Ft^2-Sec. and corresponding total stream enthalpies of 5000, 10,000, and 17,500 Btu/Lb. Test measurements confirmed the expected reduction in downstream heat transfer and ablation resulting from upstream mass injection. Downstream heat transfer rates determined from models having "THERMO-LAG" leading edges were 25 to 45 percent less than the theoretical rate.

INFRARED RADIATION TESTS

Twenty-five radiation-ablation tests of "THERMO-LAG" T-500 material were performed at cold wall heat fluxes ranging from 5 to 30 Btu/Ft^2-Sec. Cylindrical "THERMO-LAG" T-500 models were fabricated instrumented with four thermocouples at varying and known depths in the material. Measurements were made for the purpose of establishing temperature-time derivatives required to obtain parameters for use in the numerical ablation program. These parameters were:

1. The specific energy of chemical reaction of gaseous ablation products within the debris layer.
2. Heat capacity term for the gaseous ablation products.
3. Effective thermal conductivity of the debris.

The results of the tests and analysis are shown in Figure 6.

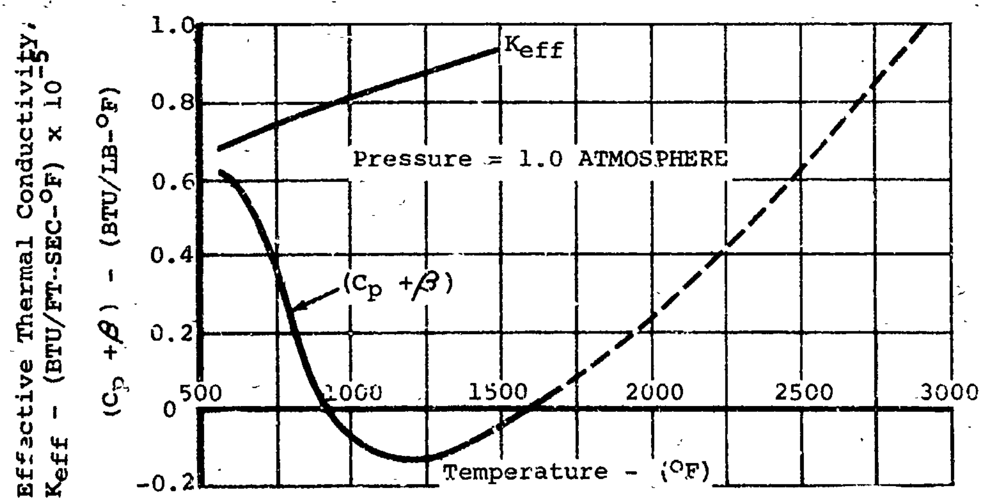

Figure 6. Effective Thermal Conductivity of the Debris Layer and the Specific Energy of the Gaseous Ablation Products.

VACUUM EXPOSURE. Specimens of "THERMO-LAG" T-500 material were exposed to pressure in the range of 10^{-4} Torr for extended times to determine material weight loss at temperatures ranging from 80° to 250°F. Data from the test series were analyzed and the following general conclusions were drawn:

1. The rate of weight loss decreases as vacuum exposure time increases.
2. The rate of weight loss increases with increased material temperature.
3. The rate of weight loss increases as initial specimen thickness increases up to a limiting thickness at which point the rate becomes a function of time and temperature only.

An analysis of the effect of material losses during a 14-day exposure to space environment on the design weight of a superorbital vehicle heat shield was made. To compensate for material losses, an increase in heat shield weight from 858 to 978 pounds would be required.

A numerical analysis technique was used to assess the effect on material thermal performance of selective subliming salt losses up to 20 percent. The analytical results indicate a 20 percent loss of salt to cause an 11.3 percent increase in total material consumption and a three percent increase in surface temperature during 300-second exposure to a plasma stream typified by a cold wall stagnation point heat flux of 110 Btu/Ft^2-Sec. and a total enthalpy of 15,052 Btu/Lb.

Comparative air arc tests were performed on identical test models, one of which was stored at normal atmospheric conditions for 100 hours, the other being held at 10^{-4} Torr and 250°F. No significant difference in performance was observed.

PHASE II

MECHANICAL PROPERTIES OF "THERMO-LAG" T-500 MATERIAL

CONTRACT REQUIREMENTS. Mechanical properties shall be obtained for both molded and sprayed versions of "THERMO-LAG" material. Such properties as shear and tensile strength, flexural strength and stiffness and elastic moduli shall be explored for virgin, and partially charred materials. The effect of ultra-violet radiation on flexural strength shall be explored. The boost environmental effects shall be studied by acoustic tests of "THERMO-LAG" sprayed on honeycomb sandwich support structure. In all pertinent cases, these mechanical properties shall be determined as functions of temperatures.

CONTRACT INVESTIGATIONS AND RESULTS

TENSILE STRENGTH. Twelve tensile tests were performed on each of the "THERMO-LAG" materials, T-500-4 spray deposited and T-500-6 glass reinforced and molded. Tests were performed in accordance with ASTM D759-48 at temperatures ranging from -150 to 400° F. Abridged mean results of the tests are as follows:

	T-500-4				T-500-6			
Test Temperature, °F.	-150	0	75	400	-150	0	75	400
Ultimate Tensile Strength, psi	600	865	390	20	1310	1190	820	270
Initial Modulus, psi x 10^{-3}	170	132	50	4	182	170	62	10
Elongation, Percent	0.60	0.75	4.65	3.20	0.75	1.00	1.95	2.00

SHEAR STRENGTH. Punch type shear strength tests were performed using the ASTM D732-46 procedure with "THERMO-LAG" T-500 test specimens of 0.25 inch thickness. Multiple tests were performed over the temperature range of -150° to 250° F. The abridged mean results are:

	T-500-4				T-500-6			
Test Temperature, °F.	-150	0	85	400	-150	0	85	400
Shear Strength, psi	2450	2200	700	250	2340	2000	980	250

FLEXURAL STRENGTH. The flexural properties of "THERMO-LAG" T-500 were determined in accordance with ASTM D790-61A using rectangular bars of material, 4.0" x 0.25" x 0.5". Multiple tests were performed in the -150 to 400° F. range. Abridged mean results are as follows:

Test Temperature, °F.	T-500-4				T-500-6			
	-150	0	90	400	-150	0	90	400
Flexural Strength, psi	2040	2400	1260	240	3200	4040	2060	400

ULTRA-VIOLET EXPOSURE. Flexural strength specimens were exposed to ultra-violet radiation, wavelength 2537-3650 A°., for a period of 14 days following which time room temperature flexural strength tests were performed. The test results indicate ultra-violet exposure to have negligible effect on "THERMO-LAG" T-500 flexural strength.

Test Temperature, °F.	T-500-4				T-500-6			
	-150	0	77	400	-150	0	77	400
Flexural Strength, psi	--	--	1246	--	--	--	2218	--

FLEXURAL STIFFNESS. The flexural stiffness of "THERMO-LAG" T-500 was determined in accordance with ASTM D747-61 using equipment modified to permit testing at elevated temperatures. Stiffness moduli were calculated from specimen measurements, load and rotation determinations. Satisfactory agreement with tensile moduli was obtained. Abridged results are as follows:

Test Temperature, °F.	T-500-4				T-500-6			
	-150	0	73	400	-150	0	73	400
Stiffness Moduli, psi x 10^{-3}	247	209	46	3	257	215	77	18

PHASE III

THERMAL PERFORMANCE ANALYSIS

CONTRACT REQUIREMENTS. Methods of thermal performance analysis shall be developed which, by applying the data obtained in Phase I, will enable accurate prediction of ablator performance in flight within the region of interest specified in Appendix A for afterbody considerations.

A routine shall be prepared in Fortran for 7040 or 7090 operation to analyze the system in one dimension. This shall provide for: input of convective and radiative flux, recovery enthalpy, pressure, and state of boundary layer; blocking of convective flux by transpiring gases, and of convection by hot wall effects; oxidation and erosion of surface as a function of surface temperature, with allowance for diffusion of oxygen through the boundary layer in the presence of transpiring gases; heat released by chemical reactions at the surface; reflection and reradiation at the surface; heat conduction through the carbonaceous material, and through the debris layer, and virgin subliming material; chemical kinetics in the subliming material and the heat of decomposition; conduction through the bond line and the backup structure.

CONTRACT INVESTIGATIONS AND RESULTS. In accordance with the requirements of NASA Contract NAS 9-877, Emerson has developed a computer program for the prediction of ablative material performance in a thermal environment characteristic of that defined for the APOLLO Command Module.

The total program, while specifically prepared for a one-dimensional subliming ablator model, has maintained a generality throughout by establishing all data such as radiant and convective heat flux, film coefficient, etc., as inputs to the computer. Any arbitrary number of computing points within the material and any combination of material properties may be considered. The program may be utilized to analyze material performance in rocket nozzles, combustion chambers, aerodynamic heating regions, radiant heating environments, and in solid-state heat conduction problems. The program may be used to compute the performance of charring (debris forming) and non-charring ablation materials, solid-state heat storage heat shields, and radiation cooled structures. The program is sufficiently general to allow the use of schemes such as implicit, explicit, or Crank-Nicholson numerical approximations by the designation of proper input parameters. Inputs to the program are convective and radiative heat fluxes, local enthalpy, pressure, state of the boundary layer, and thermodynamic properties of the heat shield material and substrate material, and the physical dimensions of the problem.

The program will predict material performance in terms of:

1. Virgin material recession (thickness history)
2. Debris recession
3. Total mass loss rate
4. Surface temperature
5. Backside temperature
6. Temperature at any prescribed point in the debris layer, virgin material, or substrate material

The program accounts for the following heat and mass transfer mechanisms with fluid boundary layer characteristics:

1. Sublimation of inorganic subliming compound
2. Transport of volatilized gases through porous debris matrix

3. Dissociation of sublimate gases in the porous matrix
4. Effect of mass injection into the boundary layer upon convective heating
5. Surface chemical kinetics due to oxidation
6. Radiant heat flux away from the debris surface
7. Conduction through the bond line and substrate

A program manual was prepared including detailed discussions of problem formulation, numerical techniques employed, input and output format, program stops, operating instructions, statement of the program in Fortran, and a glossary.

A heat conduction problem, a transient flight simulation ablation problem, and several steady state ablation problems were run to verify the program. Verification was accomplished by reproducing the test data with experimentally determined thermodynamic parameters.

PHASE IV

ATTACHMENT DEVELOPMENT

CONTRACT REQUIREMENTS. Techniques for bonding molded "THERMO-LAG" shall be developed and demonstrated by shear and tensile tests throughout the working range. Since a sub-structure capable of withstanding high temperatures is desirable as a backup, bonding techniques must be applicable to stainless or super-alloy materials.

Mechanical attachments with both metal and "THERMO-LAG" attachment members shall also be developed and tested in tension and shear.

The effects of the boost environment shall be studied by acoustic tests of molded panels of "THERMO-LAG" bonded and mechanically attached to honeycomb sandwich structure.

The effects of thermal cycling from -150°F to 250°F shall be determined for the various attachment techniques.

INVESTIGATIONS AND RESULTS

SURFACE TREATMENT. A study of thirteen general categories of surface treatments for the preparation of PH 15-7 Mo steel was conducted for the selection of the procedure providing superior bond strength of spray deposited "THERMO-LAG" T-500-4 to the basis metal. Based on comparative bond tension and bond shear test results, conducted over the temperature range of interest, a sulfuric acid reverse etch technique was selected. The bond strength values of spray applied T-500-4 to sulfuric acid reverse etch treated PH 15-7 Mo steel are as follows:

Test Temperature, °F	-250	0	80	300
Bond Shear Strength, psi	470	470	560	495
Bond Tensile Strength, psi	40	550	1420	250

SECONDARY ADHESIVES. Twenty-one adhesives of the silicone, urethane, phenolic, or epoxy types were evaluated for selection of the superior material for bonding "THERMO-LAG" T-500-6 to PH 15-7 Mo steel surfaces. The superior four metal surface treatment procedures used in the preceding study were tested with each candidate adhesive. Based upon bond shear and bond tensile strength tests over the temperature range of -250 to 300°F., a phenolic adhesive, HT-424, proved superior for secondary bonding applications. The adhesive material provides the following bond strengths to H_2SO_4 reverse etched PH 15-7 Mo.

Test Temperature, °F.	-250	0	77	300
Bond Shear Strength, psi	471	384	339	76
Bond Tensile Strength, psi	56	441	191	27

MECHANICAL FASTENERS. A mechanical fastener consisting of both "THERMO-LAG" and metal components was developed and tested as a means of attaching "THERMO-LAG" T-500-6 panels to a vehicle structure without destroying the integrity of the heat shield. The fastener consists of a T-500 stud assembly containing an aluminum insert in the shank portion. A 360 degree radial spring surrounds the stud transmitting the differential thermal load into the plate-nut portion of the fastener. This portion of the fastener is attached to the substructure by screws or rivets distributing the shear, torsion or overturning moments into the basic outer structure. An aluminum stud and retainer assembly secured to the internal end of the plate nut housing permits the "THERMO-LAG" stud to be secured. A nylock insert prevents loosening of the stud assembly. An exploded view of the fastener is shown in Figure 7. Fastener assemblies were subjected to shear and tensile tests at various temperatures to determine failure loads. Average test results are:

Test Temperature, °F.	-150	75	200	300
Ultimate Tensile Load, lbs.	336	281	107	126
Ultimate Shear Load, lbs.	275	136	115	115

VIBRATION. Test panels were prepared for vibration testing consisting of a 3/8 inch honeycomb sandwich substructure to which "THERMO-LAG" was applied by means of spray deposition, secondary bonding, and mechanical attachment. The direct spray and the secondary bonded panels are 2 feet square having 0.20 inch of T-500 material applied. Two 1' x 1' panels were prepared, each having four mechanical fasteners securing 0.4-inch thick T-500 in one case and 1.0 inch T-500 in the other. The panels were submitted to NASA, MSC, Houston, Texas for acoustic or mechanical vibration testing.

Tests were conducted to assess the effect of thermal cycling between -150 and 250°F. upon assemblies consisting of 10 inch diameter PH 15-7 Mo cylinders to which "THERMO-LAG" had been applied by the three attachment methods.

Figure 7 . Layout of "THERMO-LAG" T-500 Mechanical Fastener For Assembly

PHASE V

STRUCTURAL SCHEMES AND ANALYSIS

CONTRACT REQUIREMENTS. Consideration shall be given to schemes which could be used in the application of "THERMO-LAG" material to a spacecraft heat protection system. Specifically, the following problem areas should be studied and solutions suggested: joint, gap and edge fillers and sealing, hatch and window joining, field repair and maintenance and preliminary design criteria.

A simplified stress analysis shall be conducted using the thermoelastic properties of Phases I and II and assuming one dimensional heat flow.

CONTRACT INVESTIGATIONS AND RESULTS

STRUCTURAL SCHEMES. Investigations were performed as required to define the process of applying "THERMO-LAG" T-500 to a structure by means of spray deposition. The following process elements were defined:

1. Spray Equipment
2. Spraying Schedules
3. Surface Preparation
4. Curing Cycle

The importance of precise adherence to recommended procedures was emphasized by the findings of an investigation of an occurrence of blistering during arc jet exposure of several test models. The cause of the malperformance was traced to the formation of inorganic salt stratification during spray application using a non-recirculating spray apparatus.

JOINT, GAP, AND EDGE FILLER, FIELD MAINTENANCE. "THERMO-LAG" T-500 of a putty or dough consistency formulated by a reduction of volatile diluent content was investigated for use as a joint and gap filler and for field repair. A technique for establishing the degree of cure was developed based on extraction of unpolymerized solids. This technique was employed to assess the effectiveness of various means of curing the filler material. A number of chemical accelerators and combinations of accelerators were tested and were found not to provide the desired degree of cure. Conductive application of heat from a graphite heating element encapsulated in the filler material was found ineffective. A satisfactory degree of cure of filler material was obtained by application of radiant flux from an infrared source. Conditions of exposure were investigated and established for various filler material thicknesses.

STRUCTURAL ANALYSIS. Heat shields applied to a honeycomb sandwich structure were analyzed under thermal loading conditions typical of Apollo mission phases. Thermally induced loading, resulting from the very low temperatures encountered during the space transit phase, imposes the most severe demand upon the adequacy of the heat shield-substructure assembly; hence, analytical effort was concentrated on this mission phase.

Analytical methods were derived for calculating slab stresses in flat, cylindrical, and spherical panels consisting of "THERMO-LAG" bonded to or spray deposited on a honeycomb substrate. The slab stress equations are applicable at points reasonably removed from free edges and were developed for panels free of external restraints. One dimensional heat flow was assumed. Stresses as functions of Poisson's ratio, modulus of elasticity, coefficient of thermal expansion, and the temperature change from the stress free state to the temperature of interest were calculated using the field equations of elasticity. All quantities are functions of distance from the substructure-"THERMO-LAG" interface. The effects of inertia and thermoelastic dissipation were not considered in the analyses in order that uncoupled quasi-static theory could be utilized.

Study results indicate that, within the limitations of the analysis, "THERMO-LAG" T-500-6 is clearly superior to "THERMO-LAG" T-500-4. A maximum stress level of 1105 psi was calculated for the -6 material and 1170 psi for the -4 material. The stresses were computed for a large unrestrained panel consisting of a 0.75 inch thick heat shield bonded to a 2.00 inch thick honeycomb substructure subjected to a linear temperature change.

During the course of work originally contracted under NAS 9-877, a unique method of determining the thermal-structural behavior of a heat shield was developed. This method involves idealizing the heat shield as a truss network and has the advantages of theoretical simplicity and applicability to a variety of thermal-structural problems. In order to provide the NASA with comprehensive thermal-structural criteria, with particular regard to heat shield interface stresses at free edges, a contract extension was granted to refine and expand the truss analogy studies and to develop a program for rapid numerical computer problem solution.

The extended study included: (1) an investigation of the relative effects of modulus, Poisson's ratio, heat shield and bond thickness, and radii of curvature on interface thermal stress for a semi-inifinite panel bonded to a rigid substructure; (2) an analysis of the effects of thermal gradients, including the material property variations with temperature, on interface stresses; (3) study of the effects of deformation of a flexible substructure on heat shield thermal stresses; (4) investigation of interface bond thermal stresses using energy analyses of an elastic body and comparison of the truss network solution to a known classical solution; (5) experimental corroboration of the analytical results; and (6) an analysis to determine the extent to which the viscoelastic behavior of "THERMO-LAG" T-500 material should be considered.

The results of the extended analyses are as follows:

1. COMPUTER PROGRAM RESULTS

   (Task 1) A curved panel with an $R/_t$ of 3-1/3 had a 23% higher peak peeling stress and a 50% greater peak shear stress than a flat panel for a similar type problem.

   The maximum ratio of peak interface peeling stress to slab stress for a flat panel was 1.53. Poisson's ratio was 0.25 for this case.

   The maximum ratio of peak interface shear stress to slab stress for a flat panel was 1.20. Poisson's ratio was 0.50 for this case.

   For the case of plane stress, the effect of Poisson's ratio on interface stresses is not pronounced.

   (Task 3) A linear temperature variation through the heat shield reduced the peak interface peeling stress by 44 percent and the peak interface shear stress by 62 percent from that calculated for a uniform temperature. Interface stresses decreased with decreasing severity of the temperature gradient.

(Task 4) The effect of a flexible honeycomb substructure on interface thermal stresses was investigated. Again, curved panels exhibited higher peak stresses than did flat panels.

Stress ratios increased as the thickness of the substructure was increased in all cases. However, a limiting value was reached of 81 percent of the peak stress for a rigid base. This occurred for a particular face plate thickness. It would be necessary to increase the face plate thickness in addition to the substructure thickness to achieve an interface stress comparable to that for a rigid base.

2. SUBSTANTIATION OF TRUSS NETWORK

(Task 6) Energy solutions: Complementary and potential energy methods were utilized to substantiate the truss network method.

The results from a potential energy solution produced about 20% greater displacements than results from the truss network solution to an identical problem. The difference was attributed to the large grid size of the truss network which introduced constraints which restricted the displacements. This difference would be lessened with reduced grid size.

The complementary energy method was employed to verify interface stresses as calculated by the truss network method. At the forward face, the form of the stress function used in the complementary energy method imposes a value of zero shear stress regardless of applied loading. Using the truss network method, a 9000 psi shear stress at the forward face was indicated for an applied stress of 4860 psi. At distances removed from the forward face in excess of the heat shield thickness, stress distributions computed by the two methods were in close agreement.

A stress concentration factor for a plate subjected to uniform tension having a small circular hole, the diameter of which was equal to four times the grid size, was determined by the truss network method. The results were compared to a classical solution obtained from standard equations of elasticity. The truss network result was 15 percent less than the "exact" analytical solution. With reduction of the grid size, the error would be minimized.

(Task 7) A photoelastic study was conducted for the experimental determination of interface stress distributions. The results were compared with truss network solutions to similar problems. Indicated interface peeling stresses were in fair agreement along the interface with the peak stress about 20 percent less than that calculated by the truss network method. Shear stresses again differed considerably at the forward face but showed reasonable agreement along the remainder of the interface.

3. INVESTIGATION OF RATE AND TIME EFFECTS

(Task 8) At elevated temperatures, thermally induced compressive stresses in "THERMO-LAG" T-500 materials calculated by viscoelastic methods were significantly higher than those calculated by elastic methods. Relatively minor differences were found in the low temperature range with the elastically calculated stresses being somewhat higher. The study results support the conclusion that the extent of the effects of viscous behavior on structural properties should be determined for any candidate heat shield material.

PHASE VI

FABRICATION OF TEST ARTICLES

CONTRACT REQUIREMENTS. In addition to the fabrication of specimens for the testing required in the foregoing phases, at least three test panels 12 inches x 12 inches or larger, shall be made with "THERMO-LAG" material on a honeycomb substructure.

Drawings and preliminary specifications for these panels shall be supplied. Up to two of these panels shall be tested in a radiant lamp or torch facility.

CONTRACT INVESTIGATIONS AND RESULTS. Test specimens and models were fabricated from "THERMO-LAG" T-500-4 and T-500-6 for all phases of the evaluation program. Model and specimen types include:

Tab End Tensile	Flexural Strength	Plasma Jet
Standard Tensile	Flexural Stiffness	Emittance
Punch Shear	Thermal Conductivity	Thermal Expansion
Specific Heat	Vibration	Vacuum Test
Density	JANAF Tensile	Mechanical Fastener
		Infrared Radiation

PHASE VII

RECOMMENDATIONS

CONTRACT REQUIREMENTS. The contractor shall, in the final report, make recommendations to NASA for the performance enhancement, and further development and testing of "THERMO-LAG" material.

RECOMMENDATIONS. It is recommended that a program be performed to provide a thermal barrier material of the basic "THERMO-LAG" T-500 composition incorporating adjustments in material properties, particularly those affecting structural integrity of the heat shield and its ability to withstand the effects of the Apollo orbiting environment. Further, the density of the material should be adjusted to 25 to 35 pounds per cubic foot to permit a reduction in the estimated weight of the Apollo heat shield.

Pre-proposal studies of structural and thermodynamic material property allowables have been performed using analyses developed or refined under the completed contract. The comparison of structural and thermal properties shown in Table 1 presents the results of those studies.

STRUCTURAL CONSIDERATIONS. Assurance of heat shield integrity during exposure to the Apollo mission environmental sequence particularly requires a thermal barrier material capable of withstanding, without failure and with an acceptable factor of safety, repeated temperature cycling between minus 250 and 250 degrees F. Recommended material property adjustments will provide the required assurance through a reduction in the coefficient of thermal expansion, improvement of ductility or elongation capability, or compensatory trade-offs between the two properties. Basic mechanical properties such as tensile strength, bond strength, etc., will remain the same as in the present "THERMO-LAG" T-500. A comparison of present and recommended target property values is shown in Table 1.

THERMODYNAMIC CONSIDERATIONS. Desirable properties established as recommended target values based on aerothermodynamic considerations are also shown in Table 1 The reduction in density to a value of 25 to 35 pounds per cubic foot is the essential change recommended.

ENVIRONMENTAL CONSIDERATIONS. Tests performed under the completed contract have shown "THERMO-LAG" T-500 to be subject to weight losses during **exposure to space vacuum and temperature. It is recommended that studies be performed for the reduction of material weight loss in vacuum by a target value of fifty percent.**

Proposal studies of structural and thermodynamic material property allowables have been performed using analyses developed or refined under the completed contract. The comparison of structural and thermal properties shown in Table 1 presents the results of thos studies in relation to the requirements of the Apollo mission.

Property	Present "THERMO-LAG"				Target Material Properties T-500-X	
	T-500-4		T-500-6			
	Temperature-°F	Value	Temperature-°F	Value	Temperature-°F	Value
Ultimate Tensile Strength (psi)	400	20	400	250	400	250
	-150	600	-150	1300	-250	1300
Coefficient of Thermal Expansion	75 to 300	22×10^{-6}	75 to 300	12×10^{-6}	75 to 500	11×10^{-6}
Integral Average in/in-°F	75 to -150	38×10^{-6}	75 to -150	26×10^{-6}	-250 to 75	17×10^{-6}
Elongation, Percent	400	3	400	2	400	2
	-150		-150	0.7	-250	2
Modulus of Elasticity (psi)	400	3000	400	12,000	400	12,000
	-150	160,000	-150	180,000	-250	65,000
Emittance		0.9		0.9		0.9
Absorptance		0.9		0.9		0.9
Bond Tensile (psi)	300	260	300	260	400	250
	-150	125	-150	125	-250	1300
Bond Shear (psi)	300	1000	300	420	400	400
	-150	600	-150	600	-250	650
Density LB/FT^3	250	60	350	60		25-35
Virgin Material Thermal Conductivity BTU/HR-FT-°F	400	0.1135	400	0.075	400	0.07
	-150	0.0544	-150	0.055	-250	0.05
Char Thermal Conductivity	400	0.0369	400	0.038	400	0.037
	-150	0.0376	-150	0.0434	-250	0.037
Specific Heat Virgin Material	400	0.615	400	0.600	400	0.5
	-100	0.234	-100	0.200	-250	0.5
Specific Heat, Char	400	0.632	400	0.407	400	0.5
Temperature of Sublimation °F		530		530		530
Mass Transport Chemical Kinetics					Unchanged	

Table 1. Comparison of Present and Recommended "THERMO-LAG" T-500 Materials